

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

HONG *et al.*

Application No.: 10/767,854

Filed: January 30, 2004

For: Oxidation Protective Coating  
Method for Carbon/Carbon  
Composites

Confirmation No.: 2978

Art Unit: 1792

Examiner: Ngampa, Briget P.

Atty. Docket: 2236.0070000/JUK/SMW

**Declaration of Soon Hyung Hong, Yang Ho Bae, Ji Young Song and Hee  
Yeoun Kim Under 37 C.F.R. § 1.132**

*Attn: Mail Stop Amendment*

Commissioner for Patents  
PO Box 1450  
Alexandria, VA 22313-1450

Sir:

The undersigned Soon Hyung Hong, Yang Ho Bae, Ji Young Song and Hee  
Yeoun Kim declare and state as follows:

1. We are co-inventors of the invention described in one or more of the pending claims of the captioned application.
2. It is our understanding that claim 5 of the captioned application was rejected by the Examiner in the Office Action of February 11, 2008, over Galasso et al. (U.S. Pat. No. 4,425,407), Booth et al. (U.S. Pat. No. 5,330,789), Holko et al. (U.S. Pat. No. 5,021,107) and Hanzawa et al. (U.S. Pub. No. 2001/051,258).
3. It is our understanding that claim 5 as rejected by the Examiner recited: "The method of claim 1, wherein the heat-treating in (b) is performed at a temperature

- 2 -

HONG *et al.*  
Appl. No. 10/767,854

of about 1400 °C to about 1600 °C under a pressure of about 10 mTorr to about 1000 mTorr.

4. It is our understanding that the features of claim 5 have been incorporated into amended claim 1 in the present Amendment and Reply, and that claim 5 has been cancelled. Amended claim 1 recites: "A method of making an oxidation protective coating for a carbon/carbon composite, the method comprising:
  - (a) spraying a mixture comprising a vehicle liquid and Si powder on the carbon/carbon composite and drying the mixture to remove the vehicle liquid;
  - (b) heat-treating to allow the carbon/carbon composite to be impregnated with the Si, thereby forming an SiC layer and an Si layer, wherein the heat-treating is performed at a temperature of 1400°C to about 1600°C under a pressure of about 10 mTorr to about 1000 mTorr; and
  - (c) oxidizing the Si layer to form an SiO<sub>2</sub> film.
5. The temperature range of the heat treating in (b) is critical, and the considerations for the heat treating include (1) economic cost considerations, and (2) functional considerations, including the complete and uniform infiltration of the Si on the carbon/carbon composites.
6. To minimize the economic costs associated with heat treating, we have found that high temperature processing above 1600 °C is not necessary. This point was identified in the captioned application at paragraph [0044].

- 3 -

HONG *et al.*  
Appl. No. 10/767,854

7. Fluidity of Si powder is important in the process of Si infiltration. Solid state Si powder does not have good fluidity, making movement of the Si into carbon/carbon composite pores difficult. Also, in order to be coated, the carbon/carbon composite substrate and Si should react together. Reactions between the carbon/carbon composite and Si are minimal if Si is in a solid state.
8. Fluidity of Si is insufficient at 1380 °C, resulting in an imperfect coating as shown in Fig. 1.

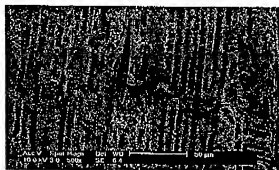


Fig.1. When infiltration is performed at a low temperature (1380 °C), carbon fiber shows and pores or cracks are not coated perfectly.

9. The known melting temperature of Si is 1412 °C. To increase the diffusion speed of the Si into the pores, the temperature should be increased near to or higher than the melting point of Si. In order to investigate temperatures which result in an effective fluidity of Si, heat treating temperatures between 1400 °C ~ 1600 °C were attempted.

- 4 -

HONG *et al.*  
Appl. No. 10/767,854

10. When the heat treating was performed near the melting point (1412 °C), Si fluidity increases as shown in Fig. 2 and the Si adheres to the carbon/carbon composite.

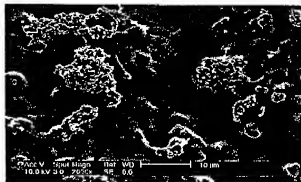


Fig. 2. Carbon/carbon composite Si-infiltrated at 1412 °C (melting point).

However, Fig. 2 demonstrates that while Si coats the composite in some areas, other areas contain pores. The pores result from the insufficient fluidity of Si, indicating that the heat treating should occur at a temperature at or above the melting point.

11. When the heat treating temperature increased to 1425°C, the carbon/carbon composite was coated uniformly as in Fig. 3.

- 5 -

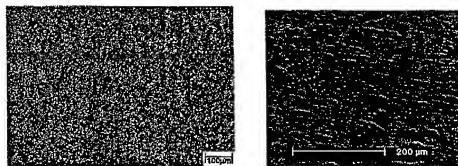
HONG *et al.*  
Appl. No. 10/767,854

Fig.3. Carbon/carbon composite Si-infiltrated at 1425 °C

The uniformly coated Si on the carbon/carbon substrate improved oxidation resistance.

12. The data presented herein demonstrates the critical nature of heat treating the Si-coated carbon/carbon composite to impregnate the initial coating layer and cracks in the initial coating later with Si.

- 6 -

HONG *et al.*  
Appl. No. 10/767,854

13. We hereby declare that all statements made herein of our own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Oct. 22. 2008  
Date

Soon Hyung Hong  
Soon Hyung Hong

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Date

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Yane Ho Ran

Oct. 22. 2008  
Date

Ji Young Song  
Ji Young Song

Oct. 22. 2008  
Date

Hee Yeoun Kim  
Hee Yeoun Kim

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- 6 -

HONG *et al.*  
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Date

Soon Hyung Hong

2008/10/23

Date

Yang Ho Bae

Date

Ji Young Song

Date

Hee Yeoun Kim

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